# NASA TECHNICAL NOTE



# **NASA TN D-5807**

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STATIC AND DYNAMIC FATIGUE BEHAVIOR OF GLASS FILAMENT-WOUND PRESSURE VESSELS AT AMBIENT AND CRYOGENIC TEMPERATURES

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION • WASHINGTON, D. C. • MAY 1970



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1.	Report No. NASA TN D-5807	2. Government Acc	ession No.	3. Re	ecipient's Catalo	g No.			
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7.	Author(s) Morgan P. Hanson		اً اِ		rforming Organiz	cation Report No.			
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12.	Sponsoring Agency Name and Address National Aeronautics and Sp		ation	T	echnical Not	e			
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15.	Supplementary Notes		. 1						
16.	Scale-model pressure vessels in the form of right circular cylinders were investigated under static and cyclic loading in ambient and liquid nitrogen environments. Tests were performed at various percents of the single-cycle burst strength to establish fatigue behavior. Liner concepts using a polyimide film, aluminum foil, and electroformed aluminum were investigated. Comparisons were made of the static and dynamic fatigue life of cylinders in ambient and liquid nitrogen environments.								
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17.	Key Words (Suggested by Author(s))  Composites Cryogenics  Unclassified - unlimited								
	Glass fibers Polymeric resins					İ			
	Pressure vessels								
19.	Security Classif. (of this report)	20. Security Class	if. (of this page)	21. 1	No. of Pages	22. Price*			
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# STATIC AND DYNAMIC FATIGUE BEHAVIOR OF GLASS FILAMENT-WOUND PRESSURE VESSELS AT AMBIENT AND CRYOGENIC TEMPERATURES

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## SUMMARY

An experimental investigation was conducted to determine the static and dynamic fatigue behavior of scale-model filament-wound glass-reinforced plastic (FRP) pressure vessels at ambient and cryogenic temperatures. At ambient temperatures, adhesively bonded polyimide-film-lined cylinders sustained cycling to failure of the FRP. Thin aluminum foil liners (0.003 in. or 0.0762 mm) sustained pressurization at ambient temperatures and in liquid nitrogen (-320° F or 77 K). An electroformed aluminum liner showed promise as a suitable liner material for use at cryogenic temperatures.

Results of static and dynamic fatigue tests of FRP cylinders indicated significant improvements of fatigue life at liquid nitrogen temperature as compared with ambient temperature. Under static loading at liquid nitrogen temperature, an FRP cylinder sustained pressurization at about 90 percent of the single-cycle burst strength for 88 days without failure of the FRP. At ambient temperature, the static life at 90 percent of the burst strength was about 7 minutes. Under cyclic loading in liquid nitrogen, no failure resulted after 1509 cycles at 55 percent of the single-cycle burst strength. Under the same cyclic loading at ambient temperature, the test results would predict failure in the FRP.

#### INTRODUCTION

Glass fibers have shown outstanding strength and strength-to-density-ratio properties that make their application to pressure vessels particularly attractive. The application has been generally in the form of filament-wound glass-reinforced plastic (FRP) (ref. 1). However, after extended periods of static or dynamic loading at ambient temperatures, the rate of strength degradation of FRP pressure vessels has been high compared with that of metallic pressure vessels (ref. 2). Under stress, the resin matrix generally cracks or crazes (refs. 3 and 4), which exposes the glass fibers to atmo-

spheric moisture and causes degradation of the fibers. At cryogenic temperatures, however, the moisture problem is reduced, and an improvement in fatigue properties would be expected. Furthermore, the glass strength increases significantly as the temperature is lowered (ref. 4).

The application of glass filament-wound pressure vessels in the containment of cryogenic liquids, however, has been limited because of liner problems; both plastic films and metallic foils have been investigated as solutions to the problem (ref. 5). Limited success has been attained because of general embrittlement of polymers at cryogenic temperatures and the strain incompatibility of metallic foil liners with the high extensibility of glass fibers (>3 percent). It has been shown, however, that thin, bonded aluminum foil liners are feasible under limited cyclic life. Usually the problem area has been in the seams required in fabrication with foil material. Limited liner life has restricted the investigation of fatigue in FRP pressure vessels. Recent developments in electroforming thin nonporous metals such as aluminum (ref. 6) show promise in fabricating liners without seams.

As a continuation of a program investigating FRP pressure vessels, model vessels were subjected to static and cyclic loading in ambient and cryogenic (liquid nitrogen) temperature environments. The performance was determined by investigating cylinders with liners of a polymeric film, aluminum foil, and electroformed aluminum.

## MATERIALS AND EXPERIMENTAL PROCEDURE

## Cylinder Fabrication

A schematic diagram of the cylinders and end caps used in the investigation is shown in figure 1. The cylinders were open-ended right circular cylinders 7.5 inches (19.1 cm)

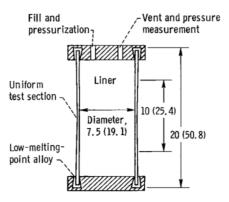


Figure 1. - Biaxial cylinder with removable end caps used for cyclic tests. (Dimensions are in inches (cm).)

in diameter by 20 inches (50.8 cm) long. Table I lists the materials used in the cylinder fabrication.

The cylinders were fabricated on mandrels of thick-wall aluminum tubing. A slight taper was provided to facilitate the removal of the finished cylinder from the mandrel. In the case of the aluminum foil liners and the polyimide film liners, a sufficient length of material was wrapped on the mandrel to allow a lap seam of about 0.25 inch (6.35 mm). The seam was made with G207 adhesive in such a manner as to assure a smooth, close fit of the liner on the mandrel. The electroformed aluminum liner was electrodeposited on an aluminum mandrel that had been first flash coated with about 0.001 inch (0.0254 mm) of silver. Electroforming parameters were controlled to produce a uniform thickness of about 0.005 inch (0.127 mm).

The same fabrication procedure of the FRP cylinders was followed for all liner concepts. The liner on the mandrel was coated with the G207 adhesive and a hoop layer of glass roving applied at a spacing of 60 ends per inch (23.6 ends/cm). The ratio of hoop to longitudinal windings was 2:1, with an arrangement of inner and outer hoop windings and a single longitudinal layer in between. A 10-inch- (25.4-cm-) long test section of uniform thickness was maintained in the center of the cylinder. The ends were reinforced with glass roving in a step arrangement to minimize the transition from the test section to the end cap restraints. The shape of the end reinforcement also provided a

TABLE I. - CYLINDER MATERIALS AND FABRICATION PARAMETERS

Roving material	S/901 single end glass <sup>a</sup>		
Roving spacing	60 ends/in. (23.6 ends/cm)		
Winding orientation	One longitudinal and two hoop layers		
Epoxy resin matrix	ERL 2256/ZZL 0820, <sup>b</sup> 27 PHR		
Liner materials	3-Mil (0.0762-cm) 1100-0 aluminum foil 3-Mil (0.0762-cm) Kapton <sup>c</sup> 5-Mil (0.127-mm) electroformed aluminum		
Liner adhesive	G207 <sup>d</sup>		
End closure seal	Cerromatrix <sup>e</sup> (room temperature tests) Cerrobend <sup>e</sup> (liquid nitrogen tests)		

<sup>&</sup>lt;sup>a</sup>Owens-Corning Fiberglas Company.

<sup>&</sup>lt;sup>b</sup>Union Carbide Epoxy Resin, Union Carbide Corporation.

<sup>&</sup>lt;sup>c</sup>E. I. duPont de Nemours & Company.

dGoodyear Aerospace Corporation.

<sup>&</sup>lt;sup>e</sup>Cerro Corporation.

means of locking the cylinder to the low-melting-point alloy in the end caps.

The filament-wound cylinders with adhesively bonded liners were removed from their mandrels by shrinking the mandrels with liquid nitrogen. This allowed the cylinders to be removed freely without damage. In the case of the electroformed aluminum liners, the silver parted from the mandrel and was removed integrally with the liner and FRP cylinder. The silver was removed by readily peeling it from the electroformed aluminum liner.

# Static and Cyclic Tests of Filament-Wound Glass-Reinforced Plastic Cylinders

Three liner concepts were used in the present investigation. For static tests in ambient and liquid nitrogen environments, thin (0.003 in. or 0.0762 mm) aluminum foil liners were used. In static testing, the cylinders were pressurized to predetermined percents of the single-cycle burst strength with a constant-pressure source. Ambient tests were conducted using oil as the pressurizing medium. Static tests at -320° F (77 K) were conducted in a cryostat in which the FRP cylinders were submerged in liquid nitrogen. The cylinders were pressurized with nitrogen gas.

In the cyclic tests, the FRP cylinders tested at ambient temperature were lined with

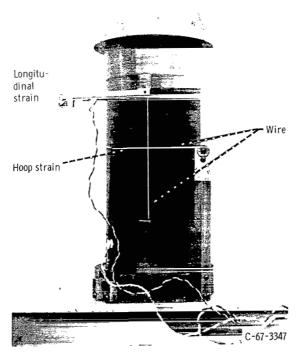


Figure 2. - Test cylinder with longitudinal and hoop strain measuring instrumentation.



polyimide film (0.003 in. or 0.0762 mm). The cylinders were pressurized with oil and cycled at a rate of 2 cycles per minute. In liquid nitrogen testing, the cyclic test was performed in a liquid nitrogen cryostat, and again the cylinder was submerged in liquid nitrogen. A liquid nitrogen pump was used for pressurization. The cyclic rate was also 2 cycles per minute. In both the ambient and cryogenic cyclic testing, the pressure ranged from a low of about 50 psi  $(34.5 \text{ N/cm}^2)$  to a maximum, depending on the percent of burst pressure required for the particular tests.

Hoop and longitudinal strains were measured by means of strain-gage deflection transducers. The hoop strain was sensed by means of a 10-mil (0.25-mm) wire mounted circumferentially on the cylinder at the midpoint of the test section. The longitudinal strain was measured similarly between clips adhesively bonded to the cylinder wall. An installation is shown in figure 2.

## RESULTS AND DISCUSSION

## Single-Cycle Burst Strength

Burst tests were performed on FRP cylinders to establish the ultimate strengths at ambient and liquid nitrogen temperatures. Table II lists the burst pressures at the test temperatures. Based on the cross-sectional area of the fibers in the hoop direction and the average burst pressure, the average tensile strength of the glass fibers was 457 000 psi (329 000 N/cm²) at ambient temperature and 625 000 psi (431 000 N/cm²) at liquid nitrogen temperature. In these strength determinations, the contributions of the resin, transverse fiber, and the liner to the strength were not included. An approximation of these factors would reduce the stress values on the order of 10 percent. The high strength is in agreement with the filament strength determined of S/901 glass in Naval Ordnance Laboratory rings (ref. 7). Also, the thin wall construction (about 0.035 in. or 0.888 mm) resulted essentially in a uniform tensile load throughout the wall thickness. The increase in tensile strength of about 36 percent from ambient to liquid nitrogen temperature agrees with that reported in the literature for S/901 glass (ref. 5).

# Static Fatigue of Filament-Wound Glass-Reinforced Plastic

The degradation of glass fibers in ambient environments has been reviewed in the literature (ref. 2). Several investigators have reported studies of stress corrosion on glass exposed to water vapor. In the present investigation, tests were performed on aluminum-foil-lined FRP cylinders to establish the static fatigue behavior. Figure 3 shows the results of sustained pressurization at various percents of the single-cycle burst strength as a function of the time to failure. Ambient temperature tests were

#### TABLE II. - SINGLE-CYCLE BURST TESTS OF

#### FILAMENT-WOUND GLASS-REINFORCED

#### PLASTIC CYLINDERS

Temperature	Burst pressure		
	psig	N/cm <sup>2</sup> gage	
Ambient	320	221	
	275	190	
	310	214	
	290	200	
	340	234	
	308	213	
	308	213	
	Average <sup>a</sup> 307	212	
Liquid nitrogen	430	297	
	410	283	
	425	293	
	415	289	
	Average b420	290	

<sup>&</sup>lt;sup>a</sup>Calculated hoop fiber stress, 457 000 psi  $(329\ 000\ N/cm^2)$ .

 $<sup>^{</sup>m b}$ Calculated hoop fiber stress, 625 000 psi (431 000 N/cm<sup>2</sup>).

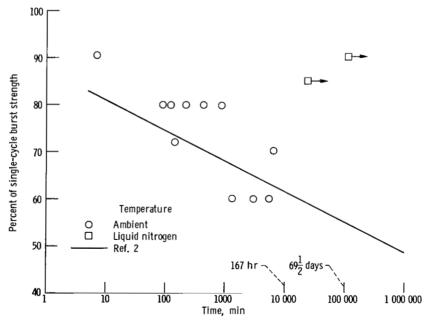


Figure 3. - Time to failure under sustained pressurization at various percents of single-cycle burst strength of aluminum-foil-lined FRP cylinders.

made ranging from 60 to 90 percent of the single-cycle burst strength. The results are compared with data from reference 2. Although scatter appears in the data of the present investigation, the general trend agrees favorably with the referenced results. Some of the scatter can be attributed to the variation in relative humidity and temperature that occurred during the testing period. At liquid nitrogen temperature where moisture is excluded, the only influencing physical factor should be the temperature because of the inertness of the nitrogen. In the two tests at liquid nitrogen temperature, no failures resulted in the FRP because of extended pressurization. Both tests were terminated because the test equipment malfunctioned. One cylinder sustained essentially constant pressurization at about 90 percent of the single-cycle burst strength for 88 days. The intent was to maintain a stable pressure in the liquid nitrogen environment. Maintaining this condition was difficult because of the problem in controlling the transfer of liquid nitrogen and pressurization with gaseous nitrogen. Figure 4 shows a continuous record of the temperature and percent of burst pressure of the cylinder pressurized at a nominal 90 percent of burst. After initial condensation of gaseous nitrogen and temperature stabilization, it was apparent that the system was not pressure tight, and, in one instance, a complete depressurization occurred. A preliminary pressure check of the cylinder at ambient temperature, however, disclosed no leaks in the cylinder. In two instances, the loss of liquid nitrogen caused the temperature to increase. Corrective

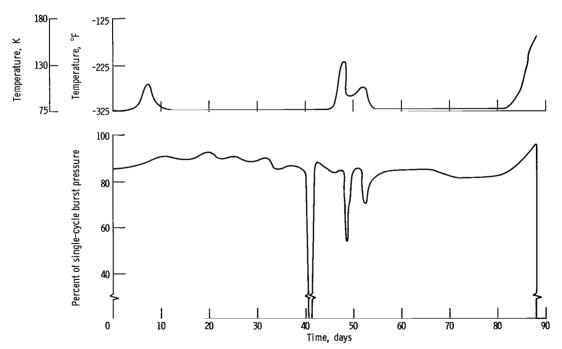


Figure 4. - Pressure and temperature profiles of static fatigue test in liquid nitrogen environment as function of time.

measures were taken, and the liquid nitrogen temperature was restored. The termination of both static tests in liquid nitrogen was the result of the cylinders bursting during unattended periods. The temperature-pressure record of one cylinder shows that a pressure rise resulted from the loss of liquid nitrogen. Although both tests were inconclusive in terms of static fatigue at liquid nitrogen temperature, both cylinders showed a significant improvement compared with the ambient static fatigue characteristics.

During the static loading of FRP cylinders at ambient temperature, hoop and longitudinal strains were recorded for a limited number of cylinders during the period of sustained pressurization. Although the cylinders were wound to have a balanced 1:1 ratio of hoop to longitudinal strain, the hoop strain was generally higher. The hoop strain as a function of time is shown in figure 5 for cylinders pressurized at various percents of

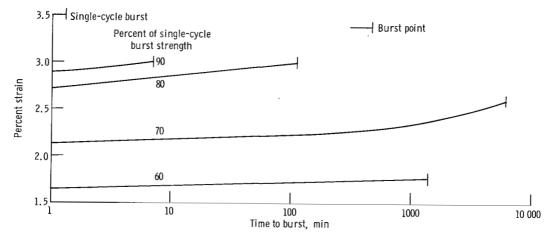


Figure 5. - Ambient temperature strain-time curves of aluminum-foil-lined FRP cylinders pressurized to various percents of single-cycle burst strength.

burst strength. The strain behavior is inconclusive because of variations in the results. However, it is interesting to observe that the static strain did not approach or exceed that of the short-time burst tests. Generally, it appears that no appreciable change in strain occurs above that of the initial strain. This has been observed in glass composites by other investigators (ref. 8).

# Cyclic Characteristics of Filament-Wound Glass-Reinforced Plastic Cylinders

The ability to cyclically test FRP cylinders is dependent on the performance of the liner. At ambient temperature, polyimide liners were adequate to allow cycling of FRP cylinders to failure. At cryogenic temperature, polyimide liners were eliminated be-

cause of questionable reliability (refs. 5 and 9).

Attempts to cycle aluminum-foil-lined FRP cylinders to failure in liquid nitrogen were unsuccessful because of limited liner life. The seam area buckled under cyclic load and caused holes in the liner. A typical buckling failure is seen in figure 6. The seam problem resulting from the foil liner construction was eliminated by lining a cylinder with one electroformed aluminum liner, as described in the Materials and Experimental Procedure section.

The results of cycling FRP cylinders at ambient and liquid nitrogen temperatures at various percents of the single-cycle burst strength are shown in figure 7. Polyimide-lined cylinders cycled to failure at ambient temperature show good agreement with pub-

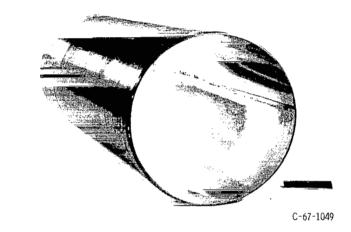


Figure 6. - Tyical seam failure of aluminum foil liner.

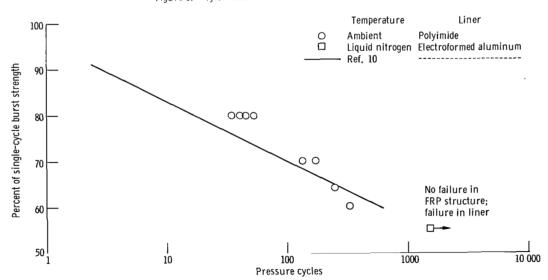


Figure 7. - Pressure cycles to failure at various percents of single-cycle burst strength of FRP cylinders with polyimide liners at ambient temperature and electroformed aluminum liners at liquid nitrogen temperature. Cyclic rate, 2 cycles per minute.

lished data (ref. 10). In liquid nitrogen, the electroformed-aluminum-lined cylinder was cycled 1509 cycles at 55 percent of the liquid nitrogen burst pressure (approx 2.1 percent hoop strain) without failure to the FRP. However, extrapolation of the ambient temperature test results to the 55-percent level would predict failure in the FRP at ambient temperature.

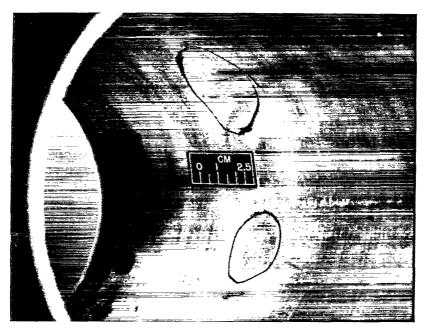
Within a few cycles of the termination of the liquid nitrogen cyclic test, it was observed that the constant cyclic rate of 2 cycles per minute was being extended. After 1509 cycles, the pump capacity could not maintain pressurization. Inspection of the cylinder after failure revealed no buckling or release by buckling of the liner from the FRP cylinder wall. Inspection by means of a fluorescent penetrant, however, revealed cracks in the aluminum. A photograph of the failed liner in place in the cylinder is shown in figure 8(a). A portion of the liner that was removed from the cylinder is shown in figure 8(b). The sample has been spread slightly to reveal the extensive fatigue cracking. The direction of the cracking is primarily transverse to the hoop direction, indicating that the maximum principal stress in the liner was in the hoop direction. However, crack deviations from the longitudinal axis indicate the influence of a biaxial stress state.

Figure 9(a) substantiates the conclusion that the liner failure resulted from fatigue. A scanning electron micrograph of the fatigue fracture surface reveals striations as a result of the crack propagation through the thickness of the liner. Surfaces of tensile fractures of electroformed aluminum (fig. 9(b)) and of wrought aluminum foil (fig. 9(c)) show different fracture phenomena.

The electroformed aluminum liner and a portion of the FRP cylinder wall are shown in figure 10(a). A fatigue crack extending through the liner is shown with no evidence of propagation into the adjoining resin adhesive. It is also interesting to note the crack in the resin matrix that terminates in the crossply fibers.

In adhesive bonding, the adherends are usually abraded or etched to enhance bonding. The electroformed aluminum appears to form an ideal surface for bonding. Figure 10(b) presents a scanning electron micrograph of the deposition surface showing an irregular surface consisting of an array of nodular asperities. Since this surface becomes the bonding surface as a result of the present fabrication process, it can be reasoned that this improved the integrity of the bond.

The increased cyclic life of the electroformed aluminum liner over that of aluminum foil liners with seams shows promise of improved reliability of the metallic liner concept for FRP pressure vessels. Fatigue life of metallic liners may limit cyclic capability but may be adequate for certain applications where the high specific strength of glass fibers must be utilized.



(a) Fluorescent penetrant examination of fatigue cracks of electroformed aluminum liner attached to FRP cylinder.



(b) Section of electroformed aluminum liner removed from cylinder and expanded to show fatigue crack patterns.  $\,$  X10.

Figure 8. - Electroformed aluminum liner as fatigued by pressure cycling in liquid nitrogen.



(a) Fatigue fracture surface of electroformed aluminum liner material.  $\times 1200$ .

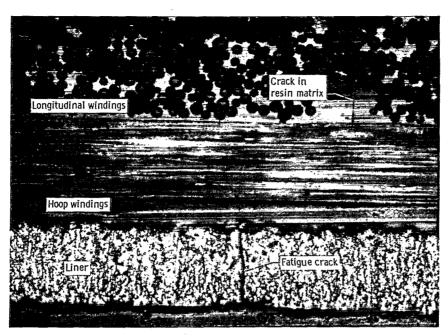


(b) Tensile fracture surface of electroformed aluminum liner material.  $\times 2000.$ 

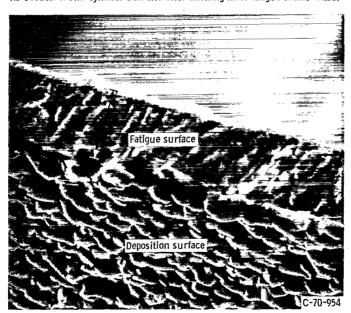


(c) Tensile fracture surface of wrought aluminum foil material, X2000.

Figure 9. - Scanning electron micrographs of fracture surfaces of electroformed aluminum and wrought aluminum foil.



(a) Section of FRP cylinder wall and liner showing liner fatigue crack. X250.



(b) Fatigue and deposition surfaces of liner material. X500.

Figure 10. - Electroformed aluminum liner material used as liner in FRP cylinder.

#### CONCLUDING REMARKS

In an investigation to determine the static and dynamic fatigue of filament-wound glass-reinforced plastic (FRP) pressure vessels at ambient and cryogenic temperatures, the static fatigue problem did not appear to be critical at cryogenic temperatures. Under static loading at liquid nitrogen temperature, an FRP cylinder sustained pressurization at about 90 percent of the single-cycle burst strength for 88 days without failure to the FRP. At ambient temperature, the static life at 90 percent of the burst strength was about 7 minutes. Under cyclic loading in liquid nitrogen, no failure resulted after 1509 cycles at 55 percent of the single-cycle burst strength. Under the same cyclic loading at ambient temperature, the test results would predict failure in the FRP.

At ambient temperatures, adhesively bonded polyimide-film-lined cylinders sustained cycling to failure of the FRP. Aluminum foil liners performed suitably in sustained pressurization at ambient temperature and in liquid nitrogen. An electroformed aluminum liner showed improvement as a suitable liner material under cyclic loading at cryogenic temperatures. Fatigue failure of the electroformed aluminum liner, however, before fatigue failure of the FRP cylinder, indicated a limitation of the metallic liner concept. Nevertheless, the increased cyclic capability of the electroformed aluminum liner over that of aluminum foil liners with seams shows promise of improved reliability. These results indicate that electroformed aluminum liners may be suitable in pressure vessel applications that require only a limited cyclic life.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, April 2, 1970,
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